Uniaxial pressure dependence of T_c from high-resolution dilatometry of untwinned $La_{2-x}Sr_xCuO_4$ single crystals

Frank Gugenberger, Christoph Meingast, Georg Roth, Kai Grube, Volker Breit, Thomas Weber, and Helmut Wühl
Institut für Nukleare Festkörperphysik and Institut für Technische Physik, Kernforschungszentrum Karlsruhe, P.O. Box 3640, 76021 Karlsruhe, Germany

S. Uchida and Y. Nakamura
Superconductor Research Course and Department of Applied Physics, University of Tokyo, Bunkyo-Ku, Tokyo 113, Japan

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We have carried out high-resolution dilatometry experiments on a series of high-quality single crystals of $La_{2-x}Sr_xCuO_4$ with various Sr content on both the under- and overdoped side of the $T_c(x)$ phase diagram. A simple *in situ* technique was applied to prevent the usual twinning of the samples in the low-temperature orthorhombic (LTO) phase, which allows us to study the full anisotropy of the thermal expansion of the LTO phase. From the anomalies in the linear expansivities $\Delta\alpha_i$ $(i=a,b,c)$ at T_c we deduce all three uniaxial stress (dT_c/dp_i) and strain $(dT_c/d\varepsilon_i)$ dependences, which turn out to be large and almost cancel for hydrostatic pressure.

Much experimental and theoretical work has been carried out in order to study the correlation between structure and superconductivity of CuO-based high-temperature superconductors (HTSC). Of particular interest are the doped lanthanum cuprates $La_{2-x}M_xCuO_4$ (M = Sr, Ba) because these compounds exhibit a variety of phase transitions at low temperatures.[1] Both the Sr-doped (LSCO) and Ba-doped (LBCO) compounds undergo a second-order structural phase transition from a high-temperature tetragonal (HTT) to a low-temperature orthorhombic (LTO) phase, where the CuO_6 octahedra tilt around an $\langle 110\rangle_{tetr}$ direction of the tetragonal unit cell. Corner-shared octahedra tilt in opposing directions. The transition temperature T_{TO} decreases monotonically with increasing x from about 500 K for the undoped La_2CuO_4 to 0 K for $x \simeq 0.22$, i.e., the tetragonal phase becomes stable for $x \gtrsim 0.22$ even at lowest temperatures. Superconductivity has been observed for $0.05 \lesssim x \lesssim 0.25$ with a maximum T_c at $x \simeq 0.15$ of about 36–38 K for LSCO (Ref. 2) and 28–30 K for LBCO.[3] The Ba-doped compound shows a strong suppression of T_c at $x \simeq \frac{1}{8}$,[3] which has been linked to another structural phase transition from the LTO to a low-temperature tetragonal (LTT) phase observed in x-ray-diffraction (XRD) measurements.[4] However, the LTO-LTT transition as well as anomalies in transport properties, e.g., resistivity,[3,5] which result from the transition, occur in a fairly wide composition range,[5,6] while the T_c suppression is strongly peaked at $x \simeq \frac{1}{8}$. It has been suggested that this suppression is caused by supercell formation in the LTT phase.[1] LSCO also shows a small dip in T_c at $x \simeq \frac{1}{8}$,[2] but the LTT phase has not been observed so far. On the other hand, a detailed study of the interplay between the occurrence of an LTT phase and superconductivity in Nd-doped LSCO revealed, that no bulk superconductivity occurs in the LTT phase and, vice versa, "the onset of superconductivity seems to inhibit a structural phase transition."[7]

The influence of the HTT-LTO phase boundary on superconductivity was investigated by Takagi *et al.* who reported a sudden drop of the Meissner fraction near $x=0.2$ for LSCO powder samples and concluded that superconductivity disappears if the system does not transform to the LTO phase.[8] This behavior was quite unexpected since the HTT-LTO transition is a second-order transition, i.e., no structural change occurs at T_{TO}, and band-structure calculations also predict little or no change of the electronic structure due to the orthorhombic distortion.[9,10] Thermal expansion results of a LSCO single crystal with $x=0.13$ measured both parallel and perpendicular to the CuO_2 planes[11] suggest that the evolution of the orthorhombic distortion is stopped by the onset of superconductivity, indicating that the orthorhombic distortion may be, in fact, detrimental for superconductivity (such behavior has been observed in A-15 compounds, for example, Nb_3Sn and V_3Si,[12] and Chevrel phases[13]). This is in accord with high-pressure experiments in which the HTT phase was stabilized at lower doping by pressure up to 2 GPa.[5] Here T_c increases linearly with pressure until the HTT phase is stabilized and then saturates. Bulk superconductivity in both tetragonal and orthorhombic phases of LSCO was also observed in subsequent studies.[14,15]

Besides the HTT-LTO transition two other structural anomalies have been observed in LSCO. Lang *et al.* investigated a series of single- and polycrystalline samples with different stoichiometry and observed structural instabilities at 36 K, i.e., at the T_c of the optimum doped material in each of the samples.[16] Therefrom the authors draw the conclusion that the maximum T_c is limited by this instability. Recently, a lattice instability was observed for a transverse elastic constant, which begins to

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soften 20 K above T_c.[17] Further, ultrasonic measurements on LSCO single crystals[18,19] revealed an anisotropic coupling of superconductivity to the lattice, i.e., the longitudinal elastic constant C_{33} softens discontinuously at T_c while for the constant C_{11} only a small slope change is seen.

In this paper we present measurements of the anisotropic thermal expansion of high-quality LSCO single crystals with $x=0.1$, 0.15, 0.2, and 0.3 between 5 and 300 K, i.e., in both the under- and overdoped regime of the $T_c(x)$-phase diagram. We have successfully applied a simple *in situ* technique to prevent the twinning of the samples in the orthorhombic phase, which allows us to study the coupling of superconductivity to the lattice in detail along all three crystallographic axes. To the best of our knowledge, these are the first measurements on untwinned LSCO crystals. We show that the evolution of the structural transition is not simply arrested at T_c, which allows us to deduce all three uniaxial stress (dT_c/dp_i) and strain $(dT_c/d\varepsilon_i)$ dependences of T_c from the measured expansivity anomalies $\Delta\alpha_i(T_c)$ using the Ehrenfest relationship. Irrespective of Sr content, one obtains a very large negative pressure dependence dT_c/dp_c for pressure perpendicular to the CuO_2 planes. The in-plane pressure effects dT_c/dp_a and dT_c/dp_b are both positive and show a pronounced anisotropy for large x.

Single crystals of LSCO with $x=0.1$, 0.15, 0.2, and 0.3 were grown by the traveling solvent floating zone (TSFZ) method (for details of experimental setup see Ref. 20). Sr content was checked by electron probe microscopic analysis with an uncertainty of $\Delta x \simeq 0.01$. The oxygen content of all samples was close to 4.0. dc magnetization experiments in a magnetic field of 10 Oe parallel to the c axis (shielding, zero-field cooled) showed almost perfect diamagnetism below T_c with sharp transitions of width less than 2 K for the superconducting samples ($T_c=28.5$, 36, and 29.5 K for $x=0.1$, 0.15, and 0.2) and no effect for $x=0.3$.[20] dc measurements of the in-plane and out-of-plane resistivity between 5 and 800 K were also carried out and the results are reported elsewhere.[20] The linear thermal expansion was measured using a high-resolution capacitance dilatometer,[21] where the temperature is varied continuously at constant rates of 10 mK/s on both cooling and heating. The actual size of the samples in these measurements was $L \approx 0.5$ mm so that relative length changes of $\Delta L/L \approx 10^{-8}-10^{-9}$ could be resolved.[21] Detwinning of the samples was achieved by applying a moderate uniaxial stress of $\sigma \approx 20-40$ MPa along one of the $\langle 110 \rangle_{\text{tetr}}$ directions *while* the crystal transforms from the HTT to the LTO structure. Since T_{TO} is below room temperature for all investigated samples the stress has to be exerted *in situ* by means of a small CuBe spring (cut from a 0.5-mm-thick CuBe sheet) in which the crystal was inserted (see inset Fig. 1).

Before we show our results a few remarks concerning the notation. The unit-cell vectors of the LTO cell are rotated by 45° around the c axis with respect to the HTT cell, i.e., $\langle 110 \rangle_{\text{tetr}} \hat{=} \langle 100 \rangle_{\text{ortho}}$. In the following we use the orthorhombic notation and omit the index "ortho."

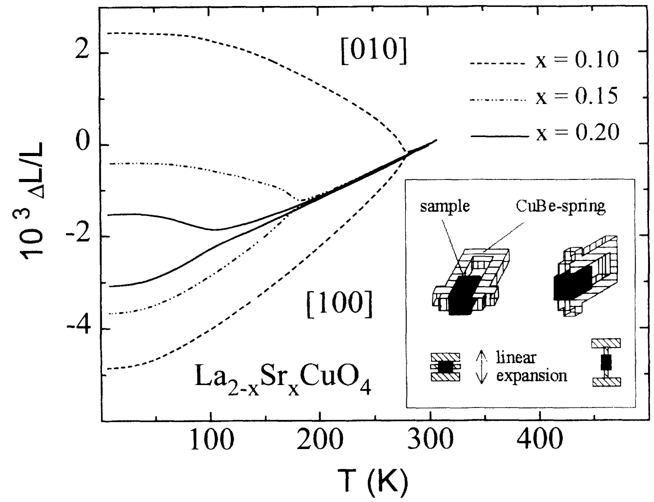

FIG. 1. Relative length change $\Delta L(T)/L_{300\text{K}}$ along the in-plane directions [100] and [010] for $x=0.1$, 0.15, and 0.2. The anisotropy below T_{TO} in the length change due to the orthorhombic distortion of the former tetragonal unit cell is clearly observed. T_{TO} decreases with increasing x, as well as the magnitude of the orthorhombicity. The inset shows how the sample is inserted into the CuBe spring. The linear thermal expansion is measured in the vertical direction as indicated by the two pistons (horizontal bars), one of them is movable and shifts a capacitor plate when the sample expands or contracts.

We arbitrarily describe the [100] direction as the a axis, since in this direction the stress is exerted by the spring and the lattice parameter in this direction becomes shorter in the LTO phase than the lattice parameter along the [010] direction $\hat{=} b$ axis as described below.

In Fig. 1, we show the linear thermal expansion $\Delta L(T)/L_{300\text{K}}$ along the in-plane directions [100] and [010] for the three superconducting samples. One observes a splitting of the linear thermal expansion below $T_{\text{TO}} \simeq 280$, 180, and 100 K for $x=0.1$, 0.15, and 0.2, respectively, which leads to an orthorhombic distortion of the former tetragonal unit cell. The magnitude of the distortion at 10 K depends on the Sr content and decreases with x since T_{TO} is shifted to lower temperatures. Our results agree well with the distortions deduced from XRD measurements[22] [e.g., 0.73% (this work) vs 0.8% (Ref. 22) for $x=0.1$] and thus we conclude that the samples are nearly single-domain crystals.

Figure 2 reviews the linear thermal expansivities $\alpha_i(T)$ along the three different crystal axes between 5 and 300 K. The onset of the orthorhombic splitting at T_{TO} appears in form of two discontinuous anomalies of opposite sign in the expansivities α_a and α_b, i.e., the jumps $\Delta\alpha_i(T_{\text{TO}}) \equiv \alpha_i(T_{\text{TO}}^-) - \alpha_i(T_{\text{TO}}^+)$ are positive for the a axis and negative for the b axis. The sharpness and the magnitude of these anomalies decrease with increasing x. The out-of-plane expansivities α_c always show positive jumps at T_{TO} due to an additional shrinking of the c axis resulting from the octahedra tilt. Since the anomalies arising from the onset of superconductivity are not well

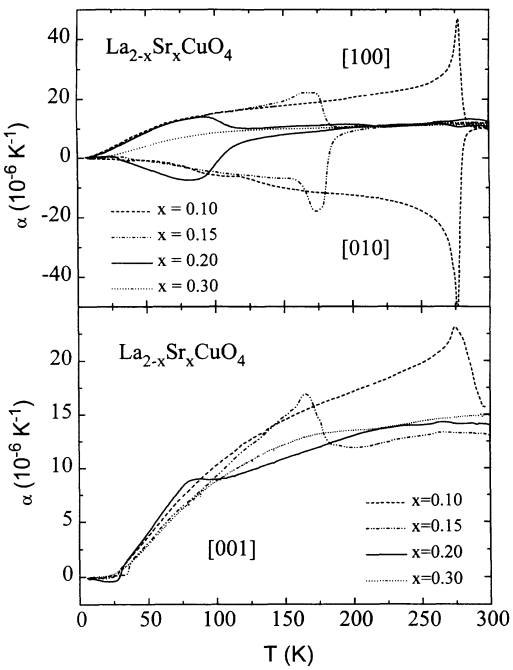

FIG. 2. Linear thermal expansivities $\alpha = d\ln L(T)/dT$ for $x=0.1$, 0.15, and 0.20 along the [100], [010], and [001] directions. For comparison we also show the expansivity of the overdoped sample ($x=0.3$), which remains tetragonal and nonsuperconducting at least down to 5 K, along the [001] and the $[100]_{\text{tetr}}$ directions.

resolved in Fig. 2, we show the expansivities in the vicinity of T_c in Figs. 3 and 4 in more detail. Figure 4 exhibits just the anomalies at T_c, which were obtained by subtracting a smooth background (approximated by a cubic spline of the data excluding a 30-K wide region centered at T_c) from the data shown in Fig. 3. From Figs. 3 and 4 it is evident that α_c exhibits large negative jumps at T_c, the magnitude of which increase with increasing x. On the other hand, the anomalies $\Delta\alpha_a$ and $\Delta\alpha_b$ are both positive for all x. While $\Delta\alpha_a$ remains small and nearly constant, $\Delta\alpha_b$ clearly increases with increasing x. The magnitudes of the $\Delta\alpha_i(T_c)$ jumps were estimated by adding an "area-conserving" idealized second-order discontinuity to the anomalies in Fig. 4 and are listed in Table I. Note that the influence of fluctuations is not analyzed. Besides the jumps at T_c and T_{TO}, no other reproducible expansion anomalies were observed in the present experiments. However, in measurements in which the crystals were not detwinned, we frequently observed instabilities between 40 and 60 K,[23] which, however, were not reproducible in detail. In one case the sample shortened a few micrometers in discrete steps in this temperature interval. The fact that these effects are seen only in twinned samples suggests that they may be related to the relaxation of twin-induced internal stresses.

The overall shapes of the $\alpha_i(T)$ curves and the anomalies at T_c for the $x=0.15$ crystal agree well with the observations of Braden *et al.*[11] for a *twinned* single crystal ($x=0.13$). Our results, however, show that the response at T_c cannot simply be explained by the freezing-in of the order parameter of the structural phase transition as suggested in Ref. 11. In such a freezing scenario [see, e.g., the behavior in *A*-15 (Ref. 12) and Chevrel[13] superconductors], the expansivity is expected to return to a value typical of the nonstructurally distorted material, which in the present case can be approximated by the expansivity of the tetragonal $x=0.3$ sample. Figure 3 clearly shows that the structural transition is not arrested at T_c since the expansivities do not jump back to the $x=0.3$ values (indicated by dashed [001] and dotted lines $[100]_{\text{tetr}}$). In fact, $\alpha_a(T_c)$ jumps away from and $\alpha_c(T_c)$ jumps beyond the expected values. In the following we calculate the uniaxial stress dependences of T_c from the expansion anomalies at T_c using the thermodynamic Ehrenfest relationship

$$\frac{dT_c}{dp_i} = V_{\text{mol}} \cdot \Delta\alpha_i(T_c) \cdot \left[\frac{\Delta C_p(T_c)}{T_c} \right]^{-1} . \tag{1}$$

As will be shown, the calculated values agree well with directly measured *c*-axis uniaxial stress experiments[24] and also with uniaxial strain dependences deduced from the

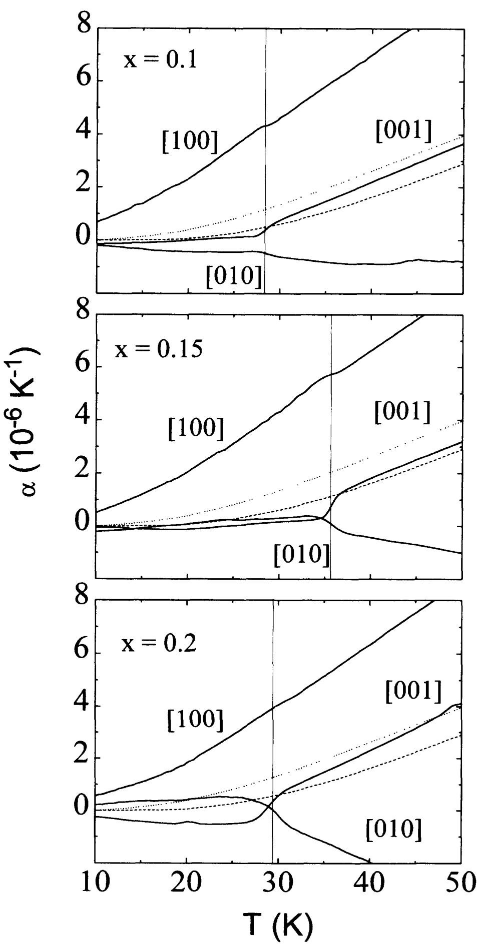

FIG. 3. Linear thermal expansivities of the superconducting samples in the vicinity of T_c, which is indicated by the vertical lines. Also shown are the expansivity of the overdoped ($x=0.3$) tetragonal sample along the [001] (dashed lines) and $[100]_{\text{tetr}}$ (dotted lines) directions.

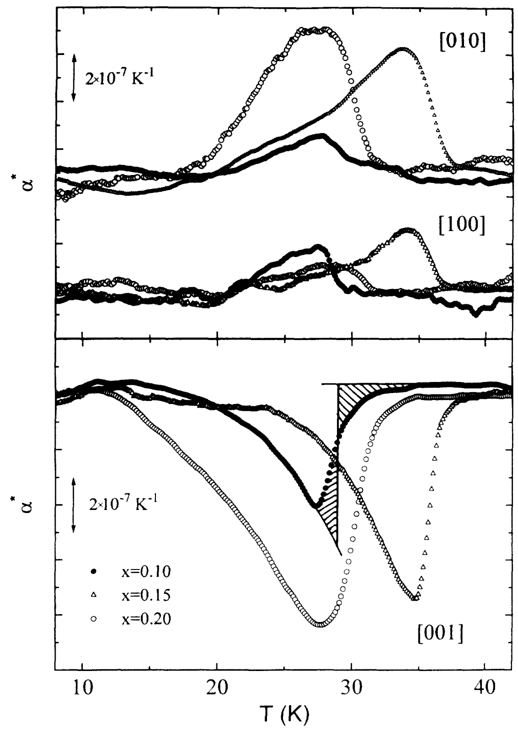

FIG. 4. Expansivity jumps $\Delta\alpha_i(T_c)$. The jumps are extracted by separating a smooth background (cubic spline) from the original data, i.e., $\alpha^* = \alpha - \alpha_{\text{spline}}$ (see text). The magnitude of $\Delta\alpha_i(T_c)$ is estimated by an idealized discontinuity and an area-conserving construction, as indicated in the figure.

ultrasound measurements.[18,19] This thermodynamic consistency provides further evidence that the expansion anomalies at T_c are not due to the freezing-in of the structural order parameter as observed in *A*-15 or Chevrel superconductors, since for these substances the Ehrenfest relationship has been shown to be invalid.[13,25]

For calculation of the uniaxial pressure effects dT_c/dp_i via Eq. (1) we need the discontinuities $\Delta C_p(T_c)/T_c$ in the

TABLE I. Estimated values of T_c, $\Delta\alpha_i(T_c)$, and $\Delta C_p(T_c)/T_c$ (from Ref. 26) and calculated uniaxial stress and strain dependences dT_c/dp_i and $dT_c/d\varepsilon_i$ for $x=0.1$, 0.15, and 0.2. For comparison, some values of $|dT_c/d\varepsilon_i|$ estimated from ultrasonic measurements (Ref. 18) are given in brackets.

x	0.1	0.15	0.2
T_c (K)	28.5±0.5	36±0.5	29.5±0.5
$\Delta\alpha_a(T_c)(10^{-7}\ \text{K}^{-1})$	3.1±0.5	3.2±0.5	1.4±0.3
$\Delta\alpha_b(T_c)(10^{-7}\ \text{K}^{-1})$	2.6±0.5	6.3±0.5	9.3±0.7
$\Delta\alpha_c(T_c)(10^{-7}\ \text{K}^{-1})$	−6.1±1.0	−8.7±1.0	−10.8±0.7
$\Delta C_p(T_c)/T_c$ (mJ/mol K^2)[a]	2.5±0.75	7.5±1.5	6.5±1.5
dT_c/dp_a (K/GPa)	7.0±3.9	2.5±0.9	1.2±0.6
dT_c/dp_b (K/GPa)	5.9±3.5	4.9±1.4	8.1±2.6
dT_c/dp_c (K/GPa)	−13.8±7.8	−6.8±2.1	−9.4±2.9
$dT_c/d\varepsilon_a$ (K)	470	250 (±340)[b]	70
$dT_c/d\varepsilon_b$ (K)	280	400 (±340)[b]	580
$dT_c/d\varepsilon_c$ (K)	−2440	−1090 (±1120)[b]	−1590

[a]Estimated from Ref. 26 (twinned samples).
[b]Estimated from Ref. 18 (twinned samples).

specific heat. Continuous heating adiabatic calorimetry on the sample with $x=0.15$ at our institute revealed a jump of 7.5±1.5 mJ/mol K^2. This value is in good agreement with data of Loram and Mirza,[26] who used a differential technique to obtain the electronic specific heat of polycrystalline LSCO samples over the whole superconducting range from $0.05 \le x \le 0.25$. Since the other single crystals are too small to be measured with our calorimeter we use the data from Ref. 26 for $x=0.1$ and 0.2.

Table I summarizes all values of $\Delta\alpha_i(T_c)$, $\Delta C_p(T_c)/T_c$, and dT_c/dp_i and Fig. 5 visualizes the anisotropy of the uniaxial stress effects. Uniaxial stress perpendicular to the CuO_2 planes is expected to strongly decrease T_c ($dT_c/dp_c \ll 0$) for all x values. This result is consistent with uniaxial stress experiments on *c*-axis-aligned grains by Motoi *et al.*[24] The in-plane pressure effects dT_c/dp_a and dT_c/dp_b are both positive. For $x=0.1$ both effects are nearly equal, but with increasing x, dT_c/dp_b increases while dT_c/dp_a decreases, i.e., the anisotropy of in-plane pressure effects is raised with higher doping. We have further calculated the uniaxial strain dependences of $dT_c/d\varepsilon_i$ which determine the change of T_c on variation of a single lattice parameter leaving all the other distances of the structure perpendicular to the strain direction unaltered. The $dT_c/d\varepsilon_i$ are given by

$$\frac{dT_c}{d\varepsilon_i} = \sum_{j=1}^{3} C_{ij}\frac{dT_c}{dp_j} , \tag{2}$$

where C_{ij} are the elastic constants. We used C_{ij} values of the (orthorhombic) undoped La_2CuO_4 compound from Ref. 27. The anisotropy of the uniaxial pressure effects is essentially reproduced in the $dT_c/d\varepsilon_i$ values (see Table I). Our uniaxial strain results are in good agreement with values deduced from ultrasonic experiments on twinned crystals[18,19] (values in parentheses in Table I), from which, however, only the magnitude (not the sign) of the uniaxial strain dependences can be determined.

There are several things worth noting concerning the uniaxial stress (strain) dependences. First, independent of

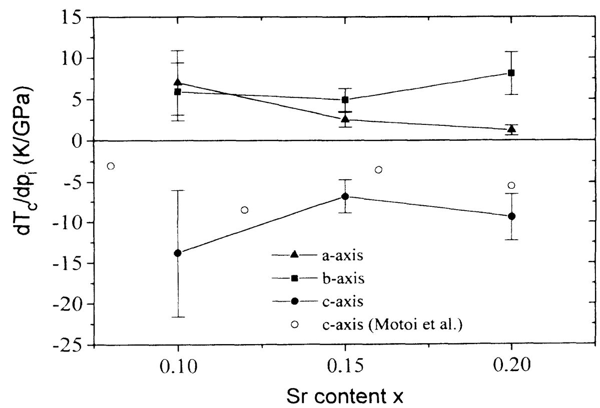

FIG. 5. Uniaxial pressure dependences dT_c/dp_i [$i=a$ (▲), b (■), and c (●)] calculated from $\Delta\alpha_i(T_c)$ via the Ehrenfest relation. For comparison we show the dT_c/dp_c from Ref. 24 (○).

the degree of doping the magnitudes of the dT_c/dp_i are very large and largely cancel for hydrostatic pressure[28,41] due to both positive and negative dT_c/dp_i terms, as was also found for optimized $YBa_2Cu_3O_x$ (YBCO).[29,30] This demonstrates that the LSCO structure is far from being optimal for a high-T_c value (this may also explain the relatively low T_c for this compound in comparison to the structurally similar (i.e., one CuO_2 plane) $Tl_2Ba_2CuO_6$ and $HgBa_2CuO_4$ structures which have much higher T_c values (≈ 90 K). Also of interest is the a, b anisotropy of dT_c/dp_i, which is largest for $x=0.2$ and absent for $x=0.1$ (see Fig. 5 and Table I). This behavior is quite unexpected since the structure becomes more isotropic (smaller orthorhombic distortion, see Fig. 1) as x increases from 0.1 to 0.2. We have no explanation for this puzzling behavior, but feel this is important and demonstrates the complexity of these materials.

There have only been a few attempts to calculate the uniaxial stress dependence of T_c of HTSC.[31–33] For LSCO ($x=0.15$) Goddard[31] predicts using the magnon pairing theory that T_c is increased by 14 K, if the a, b plane is compressed by 2% (the model considers no inplane anisotropy) and is decreased by 1 K for a 2% compression of the c axis. This agrees almost too well with our a, b results from which we calculate an increase of T_c by 13 K for 2% compression, however, the predicted negative c-axis strain effect is, although the sign is correct, by 1 order of magnitude smaller than our result.

It is instructive to compare the present results with those for YBCO, which also exhibits a maximum in T_c as a function of doping, similar to the T_c maximum in LSCO. In YBCO the pressure-induced charge-transfer model[34] has been argued to explain at least part of the pressure dependence of T_c.[29,35] In contrast to YBCO, in which T_c is raised by c-axis stress when the system is underdoped ($\delta > 0.1$) and lowered when overdoped,[35] LSCO exhibits a large and nearly composition-independent negative c-axis stress dependence. This shows that the pressure-induced charge-transfer model is not applicable for LSCO, which is not too surprising since the LSCO structure lacks the charge reservoir (chains in YBCO) from which charge can be transferred to the CuO_2 planes and, at least for hydrostatic pressure, no charge transfer has been observed experimentally.[36] This shows that a mechanism other than pressure-induced charge transfer is responsible for the large T_c changes under stress in LSCO.

Perhaps, one starting point for understanding the pressure effects in LSCO are observations from polarization-dependent x-ray-absorption (XAS) measurements[37,38] from which it has been concluded that the hole concentration on the apex oxygen sites ($O2p_z$) increases strongly above the optimum Sr concentration of $x \simeq 0.15$. This suggests that T_c decreases with increasing site population on $O2p_z$ sites supporting theoretical considerations which claim suppression of T_c by holes on apex sites.[39] One would have to prove by experiment whether a transfer of charge from the in-plane oxygen sites ($O2p_\sigma$) to the $O2p_z$ sites occurs under uniaxial pressure perpendicular to the planes.

In summary, we have measured the anisotropy of the linear thermal expansivities of untwinned LSCO single crystals in order to study the coupling of superconductivity to the orthorhombic structural distortion and to determine the uniaxial stress (strain) effects on T_c. We found that stress perpendicular to the CuO_2 planes strongly decreases T_c, while stress parallel to the planes always raises T_c. Furthermore a pronounced anisotropy evolves between dT_c/dp_a and dT_c/dp_b when the Sr content increases. At $x=0.1$ both effects are of nearly equal magnitude, but then the b-axis effect increases, while the a-axis effect decreases. Although the uniaxial stress effects are very large, they almost cancel each other, so that the hydrostatic pressure dependence is only weak. The anisotropy of the stress effects is essentially reproduced in the uniaxial strain dependences $dT_c/d\varepsilon_i$. The consistency of our calculated values with stress and ultrasonic experiments[18,19,24] makes it unlikely that the anomalies $\Delta\alpha_i(T_c)$ are substantially influenced by a coupling of superconductivity to the structural HTT-LTO phase transition (as it is the case in A-15 compounds). Only a few theoretical approaches[33–35] to describe the uniaxial stress effects on T_c in HTSC exist and none of them predicts *consistently* the very different effects in LSCO, YBCO, and $YBa_2Cu_4O_8$,[40] i.e., explains why these materials respond so differently to subtle structural deformations. We hope our experimental results will stimulate further theoretical work about the influence of uniaxial stresses and strains on superconductivity in HTSC.

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